Development of Innovative Hydrogen and Micro Energy Solutions at the Austrian Research Centers

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Hydrogen technologies are increasingly important for a number of future aerospace and terrestrial applications. The Austrian Research Centers (ARC) are presently developing an innovative hydrogen tank system based on a combination of hollow microspheres and chemical hydrides, promising very high storage densities up to 10 weight % (wt%) at ambient pressures and temperatures. ARC is also active in developing micro energy converters based on micro turbines and Stirling engines to use waste heat and exhaust for improving the overall efficiency for a number of applications. Furthermore the present paper discusses a concept that utilizes a micro energy converter together with a micro combustion module that enables a micro power generator with very high power-to-weight ratios. In addition an overview of energy-related developments is presented that is specifically suitable for aerospace and demanding terrestrial applications.

I. Introduction

The Space Propulsion & Advanced Concepts department at ARC is focusing on micro propulsion and energy systems targeting both space and terrestrial applications. Propulsion applications include both chemical and electric micropropulsion systems such as a chemical micro-rocket engine using green propellants (kerosene and hydrogen peroxide). Since a few years, our department became active in the area of innovative hydrogen solutions for future telecommunication satellites. One of the main activities there is the development of a hydrogen storage tank using hollow glass microspheres that promise storage densities approaching liquid hydrogen solutions but at ambient pressure and temperature. Recently, another hydrogen project was started looking at multifunctional structures that include innovative reversible hydrogen and oxygen tanks in combination with a fuel cell which can be used as a battery replacement that provides structural stability at the same time. Due to our propulsion expertise, we also began to investigate micro energy converters such as micro turbines and micro Stirling engines for example to harness the high energy densities produces by our micro rocket engine as a high energy-density cell. This paper will give an overview of our energy related activities as outlines above.

II. Innovative Hydrogen Solutions for Space and Terrestrial Applications

The following chapters will describe our hydrogen storage solutions based on hollow glass microspheres as well as our battery replacement - multifunctional structure using a fuel cell in combination with reversible chemical storage of hydrogen and oxygen.

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A. Hydrogen Storage using Hollow Glass Microspheres

Usually, hydrogen is stored in its liquid state requiring very low temperatures, in chemical compounds or under high pressure[1]. One possibility to store gaseous hydrogen is to use hollow glass microspheres (5 – 200 µm diameters) under high pressure (350 – 700 bar)[2]. The sphere-wall is impermeable for hydrogen at ambient temperature. The heating of the spheres increases the diffusion of hydrogen through the wall. This process requires a lot of heat thus reducing its efficiency. Furthermore the packing density of a microsphere bed is only 60 %.

Hydrogen storage in chemical compounds is presently a major research topic. For example, the reaction of sodium borohydride ($NaBH_4$) with water results in hydrogen storage capacities of about 6 wt% but on the other hand produces a lot of heat which again reduces the overall system efficiency and can be even problematic for a number of applications[3].

We developed an innovative process that combines the advantages of both hollow glass microsphere and chemical compound hydrogen storage but cancels their respective drawbacks[4,5]. The system consists of three parts (see **Figure 1**). In the first part, water is used as a functional liquid to carry the hollow glass microspheres which are loaded with up to 700 bar of hydrogen gas. The next part consists of $NaBH_4$ which is injected together with the glass microspheres into a reaction chamber. There the water reacts catalytically with the $NaBH_4$ producing hydrogen and heat. This heat is now used to release the hydrogen from the hollow glass microspheres providing a double hydrogen generation process without any external energy or heat during storage or gas release. In order to concentrate the heat to the microspheres, the spheres are coated with a catalyst increasing the overall efficiency. An important aspect is that all end products in this process are recyclable. Analysis shows that such a system can reach hydrogen storage capacities of up to 10 wt% which is considered a trigger in order to bring hydrogen solutions to automotive and other applications[6].

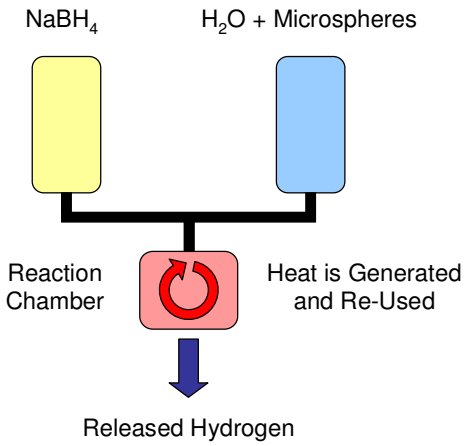

Figure 1. Schematic Process that combines both Microsphere and Chemical Hydrogen Storage

1. The Process

The hollow microspheres were purchased from 3M (S38) and doped with a Sol-Gel process to attach the catalyst. Titanium isopropoxide (TiPr) was used as a starting precursor for formation of TiO_2 layers on the microspheres. The formed mass was dried and then gently grained. Dried powder of glass/TiO_2 was added into the colloidal solution of the catalyst during the stirring. The obtained suspension was mixed and then the rest of the water evaporated. Finally the microspheres are surrounded by a bulky catalyzer film. The obtained powder was placed into a tube furnace and the heat is treated under Ar atmosphere. The spheres are surrounded by a thinner catalyzer film. The picture of the coated sphere is fuzzy due to the lower electrical conductivity of the catalyzer film (see **Figure 2**).

The mixing of microspheres with a stable sodium borohydride solution starts the hydrolysis reaction. Most of the $NaBH_4$ is converted at the surface of the spheres due to the catalyst. Hence the heat production has a maximum value at the surface of the spheres. The generated heat increases the diffusion rate of hydrogen through the wall. The converted sodium metaborate can be recycled to sodium borohydride and the spheres can be filled again afterwards.

The combination of both systems negotiates the disadvantages of high-pressure storage and chemical storage. Thus the disadvantages of sodium borohydride (low storage capacity and high temperature) were negotiated by the high storage capacity of microspheres and their required high temperature. Furthermore the specific disadvantages of spheres (packing density of 60 %, difficult transport of solid particles, and high heat capacity) were negotiating by using water as functional carrier liquid and $NaBH_4$ as a heat supplier.

The whole system could reach storage capacities of more than 10 wt% in theory. This value depends on the sphere dimensions and the weight of the spheres and the hydrogen pressure. Certainly other chemical materials can be used, which produce hydrogen and heat by addition of water. This includes Alanates, Boranates as well as simple hydrides.

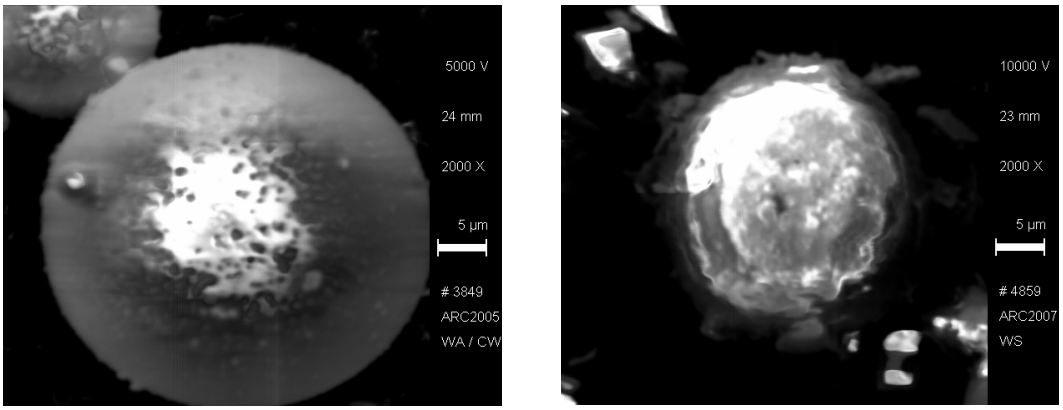

Figure 2. SEM-Picture of an uncoated (left) and a coated (right) Hollow Glass Microsphere

2. *Test Facility*

The recently established test bench is a highly flexible test bed for a convenient evaluation of various hydrogen extraction methods for microspheres and other hydrogen storage technologies (see **Figure 3**). A sophisticated control system allows a large range of variable process parameter (5 – 20 bar, 150 - 300°C, 500 – 5000 mlH_2/min). Furthermore a high speed data acquisition system facilitates in-time monitoring of all vital process parameters. A unique palladium cell is used to measure the hydrogen partial pressure and hence the released hydrogen.

3. *$NaBH_4$ Test Results*

First lab tests determined the optimum $NaBH_4$/water ratio for our test facility setup. An Erlenmeyer flask was used for these experiments. A heating unit in combination with a magnetic stirrer were used to heat and stir the mixture. It has been shown that no temperature rise was observed at ambient temperature. Pre-Heating of the system to 75°C starts the reaction. The highest temperature (ΔT = 35°C) was achieved with high amount of sodium borohydride and small amount of water. Higher temperatures can not be achieved due to the open system.

Figure 3. Hydrogen Gas Storage Test Facility

Acetic acid reacts with sodium borohydride and water to sodium acetate (CH_3COONa), diborane (B_2H_6) and hydrogen. The addition of acetic acid decreases the pH and hence accelerates the hydrolysis reaction. The highest temperature rise (ΔT = 65°C) in the open system was achieved with the addition of 4 ml acetic acid to 10 g $NaBH_4$ and 6 ml H_2O.

Venpure is a borohydride reducing agent used in organic synthesis processes and is supplied in the form of a stabilized water solution containing 12 % $NaBH_4$ and a minimum of 20 % Sodium Hydroxide (NaOH), which assures its storage stability.

The addition of acetic acid to a Venpure solution in 4 steps into an open system could not reach the temperature which could be obtained by addition of acetic acid in one step. The addition of some small amounts of catalyzer 1 (Rh/TiO_2 – glass microspheres) and catalyzer 2 (Rh/TiO_2 – glass microspheres + baking at 500°C) showed none relevant changes.

A first pre-test with 73 g $NaBH_4$ and 74 ml water was done in a closed system with external heating to 120°C. **Figure 4** shows that the hydrolysis reaction is self-sustaining and extremely exothermic at 120°C. The maximum temperature was 670°C at a maximum pressure of more than 25 bar (due to pressure measurement limitation of 25 bar). A very high temperature rise (ΔT = 550°C) in short time (< 5 s) was achieved, that means that the system is uncontrollable.

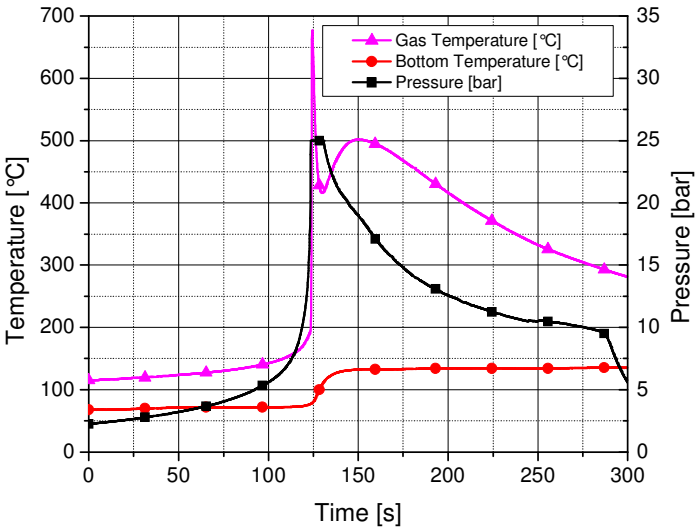

Figure 4. Pre-Test with $NaBH_4$ and Water

In comparison to the pre-test with $NaBH_4$ and water some tests with Venpure and acetic acid were done. The maximum temperature was 152°C at 6.7 bar with a high liquid-temperature and a low gas temperature. **Figure 5** shows that this system is controllable. Another test with Venpure, acetic acid and preheating was done to demonstrate the possible temperature control by chemical reaction injection. 40 ml Venpure and 30 ml water were injected into a hot chamber (preheating to 150°C). Small amounts of Venpure and acetic acid were injected once the temperature has fallen below 150°C. The total mass was 15.2 l at a maximum liquid temperature of about 170°C. **Figure 6** shows the constant temperature control by Venpure/acetic acid injection over 15 minutes.

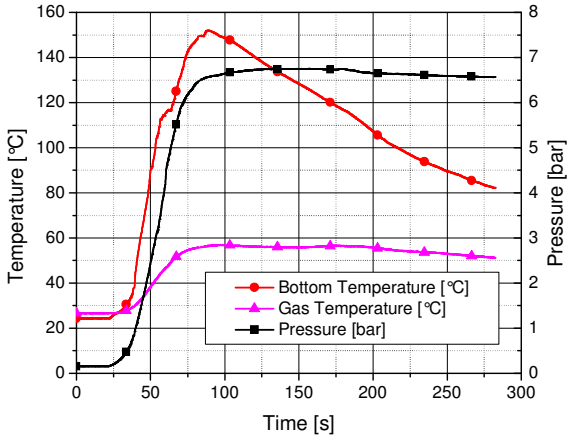

Figure 5. Test with Venpure and Acetic Acid

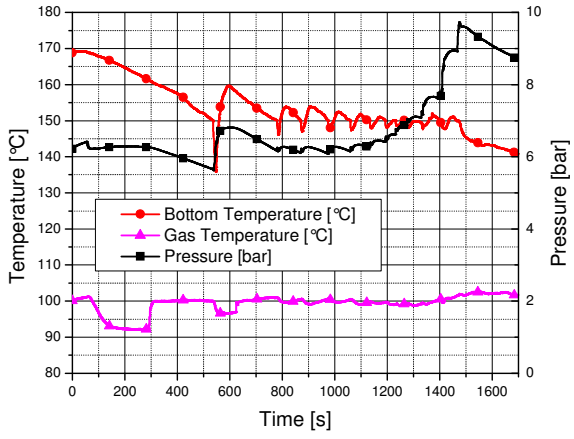

Figure 6. Injection Test with Venpure and Acetic Acid

It has been shown that injection of Venpure and acetic acid can hold the temperature at a constant level. Different parameters (ratio, start temperature, injection, mixing) influence this temperature control system. More tests are necessary to understand and optimize the system.

The achievements with respect to the $NaBH_4$ part of our system can be summarized as follows:

- Controllable reaction with Venpure
- Reaction Temperature of 150 – 170°C possible with acetic acid injection
- 55 % conversion rate of Venpure/acetic acid at RT without catalyst
- ~ 75 % conversion rate of Venpure/acetic acid at 120°C without catalyst
- 85 % conversion rate of Venpure/catalyst at 50°C (3.8 g catalyst, 40 ml Venpure)

4. Microsphere Test Results

Filling of the microspheres was done at ET EnergieTechnologie GmbH Munich due to their extensive experience in testing of hydrogen storage technologies and filling of microspheres. The filling process was done during 2 weeks in a high pressure autoclave (250°C, 700 bar, 350 h total time at maximum pressure) with an inlet diameter of about 150mm and an inlet depth of about 800 mm. First the vessel containing the microspheres was mounted into the high pressure autoclave. Then the system was heated up in several steps to the requested temperate level of 250°C. The process of pressurization was initiated subsequently until the maximum pressure level of 700 bar was reached. The actual filling phase then lasted for 350 h, before the heating system was turned off. During the next three days before the dismantling of the set-up the system temperature dropped to approx. 50°C, while the system pressure was lowered to approx. 450 bar, due to the cooling process. **Figure 7** shows the high pressure autoclave and the mounting of the transportation chamber into the autoclave.

Figure 7. High Pressure Autoclave and Mounting of the Transportation Chamber

The permeation of hydrogen through the wall depends on the hydrogen diffusivity, which are a function of the temperature and the composition of the sphere material. Furthermore the wall thickness, the pressure difference and the exchange area plays a major role. **Figure 8** presents some heating tests with filled microspheres (350 bar and 700 bar) at different temperatures. The filled microspheres with 700 bar have an age of 100 days. The released hydrogen was nearly the same for both microspheres due to the fact that approximately half of the 700 bar microspheres were broken after 100 days. This corresponds with the isostatic crush strength of the microspheres (minimum fractional survival of 80 % at 385 bar). It has been shown that a heating temperature of 150°C is ideal for microsphere gas release. Higher temperatures are not significant due to the fact that 190°C is the limit temperature for a hydride reaction based on stable sodium borohydride solutions.

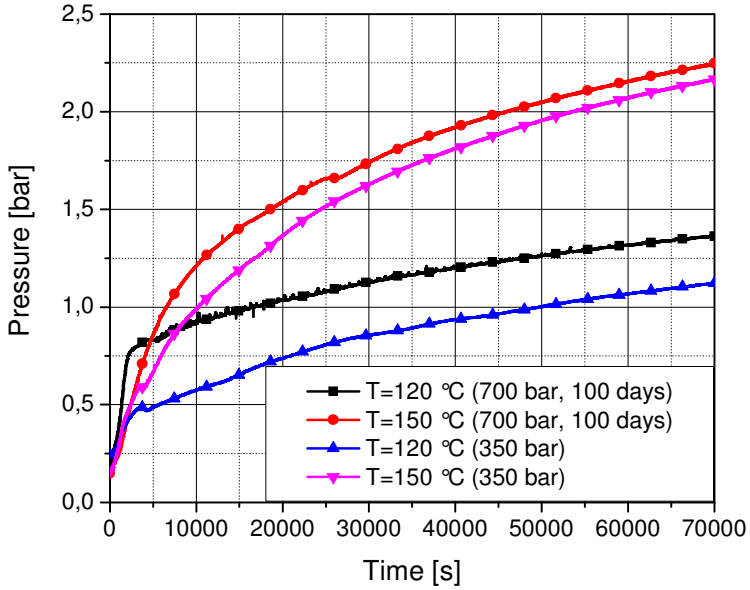

Figure 8. Heating Test with Filled Microspheres (350 bar and 700 bar)

First results are available from combined Venpure/coated microspheres tests. The tests were done with Venpure (12 % $NaBH_4$), the combined injection of Venpure and acetic acid, the combined injection of Venpure and acetic acid plus empty and uncoated microspheres (3M S38) and coated and filled microspheres (350 bar Hydrogen, 100 days old). **Figure 9** and **Figure 10** show the temperature and pressure profile of different reaction tests with a starting temperature of 120°C. Venpure injection into the hot chamber shows non relevant effects.

The addition of acetic acid increases the temperature up to 22°C. The addition of uncoated microspheres prohibited the temperature increase in the first instance due to the addition of extra mass (30 g glass microspheres). A temperature rise up to 26°C was achieved with coated microspheres. The pressure profile shows starting of the hydrogen diffusion for the filled microspheres after the Venpure and acetic acid injection. It has to be noted that some of the microspheres were broken during the injection. This effect is based on the relative low crush strength of the microspheres. **Figure 11** and **Figure 12** show the temperature and pressure profiles with a starting temperature of 150°C. A temperature rise up to 33°C and a maximum pressure of 9.3 bar was achieved with coated microspheres.

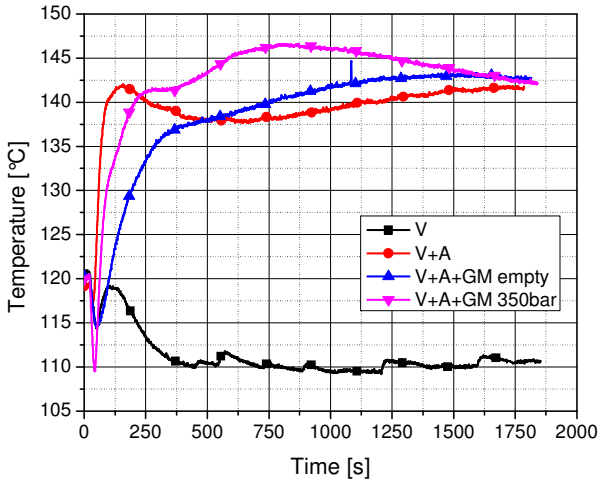

Figure 9. Temperature Profile at 120°C
(V=Venpure, A=Acetic Acid, GM=Glas Microspheres)

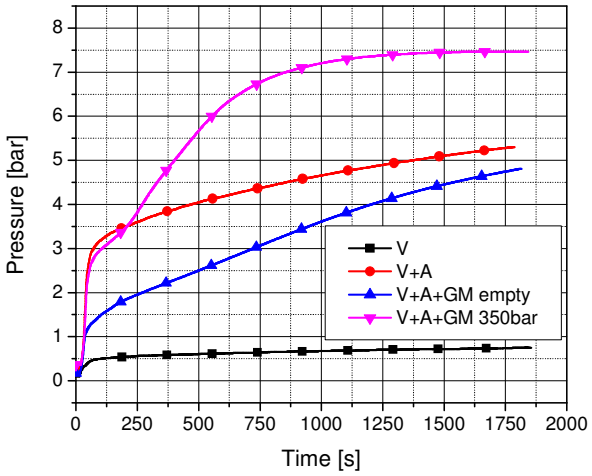

Figure 10. Pressure Profile at 120°C
(V=Venpure, A=Acetic Acid, GM=Glas Microspheres)

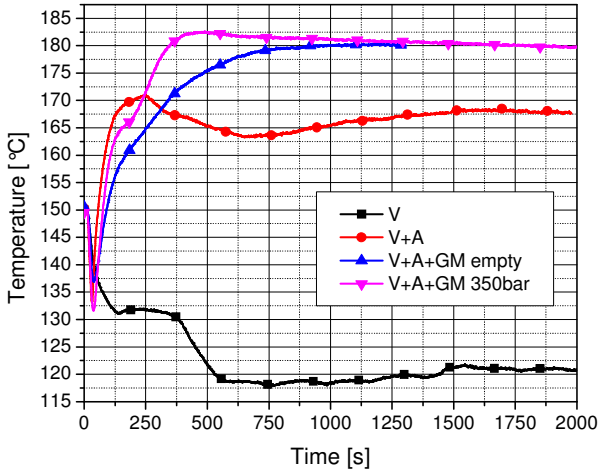

Figure 11. Temperature Profile at 150°C
(V=Venpure, A=Acetic Acid, GM=Glas Microspheres)

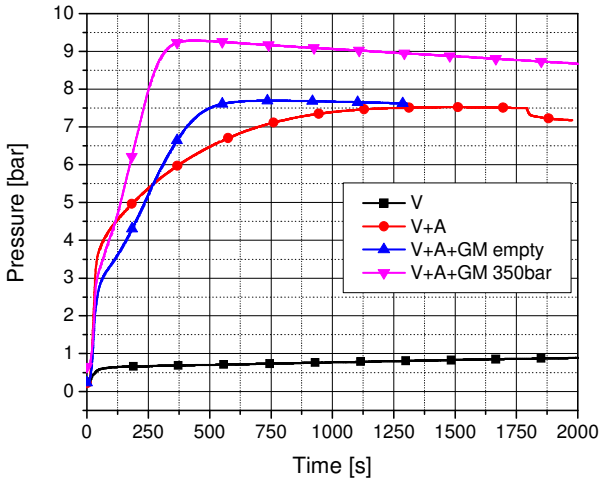

Figure 12. Pressure Profile at 150°C
(V=Venpure, A=Acetic Acid, GM=Glas Microspheres)

It has been shown that high temperatures (up to 150°C) are required for a high conversion rate of Venpure and high diffusion rate of filled microspheres. For example the conversion rate is less than 5 % at RT and less than 20 % at 120°C. Our first test results regarding catalytic $NaBH_4$ reaction correspond with the results from *Shafirovich et al*[7]. The maximum hydrogen storage capacity was **1.6 wt%** at 120°C and **2.0 wt%** at 150°C. Improvements have to be done to increase the storage capacity up to the theoretical maximum of 10 wt%.

The next steps are to test different catalysts, high strength microspheres and alternative chemicals (alanates, borates, amides, borate- and amides-mixtures, and sodium based alloys). Furthermore the prototype has to be scale-down for exact measurements and efficient thermal management

Such an innovative hydrogen storage tank could have following benefits:

- High Energy Density Low Cost Storage System
- Storage at Ambient Pressure and Temperature
- Storage Capacities of **10 wt%**
- „Free-Form Tank“
- Long-Time Storage (>1 Year)
- End Products can be recycled

Hydrogen storage and production on demand is the key for a widespread use of fuel cells. The current storage capacity of gaseous or liquid hydrogen storage systems is 6 – 10 wt%, but the specific drawbacks of each system (safety issues, energy requirements, cost, and long-term storage) prevent the introduction of hydrogen technology in several areas. The combination of ARC's microspheres and chemical hydrides has the potential to overcome these drawbacks and hence take over state of the art storage system in the future for aerospace and demanding terrestrial applications.

B. Multifunctional Structures with Fuel Cells as a Battery Replacement

A trade-off analysis regarding power supply on satellites was performed for ESA suggesting that fuel cells might be an interesting candidate to replace secondary batteries on satellites[8]. We decided to approach this topic by combining a fuel cell with innovative chemical hydrogen and oxygen storage as well as integrating such a system into a form that it can be used as a structural element as well. This would be very interesting in order to obtain a weight efficiency system which is especially important for space and automotive applications.

Our proposed system could replace present state-of-the-art batteries and lead to a reduction of up to 90 % of the volume and probably 70 % of weight. Combined with the ability to integrate the system into the satellite's structure, this novel approach seems promising for future telecommunication satellite platforms as well as for terrestrial applications. We selected a hydrogen storage tank based on sodium alanate[9,10] and a novel oxygen tank based on $YBaCo_4O_7$[11] developed at ARC as shown in **Figure 13**. The multifunctional structure (see **Figure 14**) also includes water tanks and a micro-fluidic system connected to the fuel cell in order to enable a truly reversible system just like a battery.

The following chapters will detail our hydrogen and oxygen tank solutions only since we will use an off-the-shelve fuel cell.

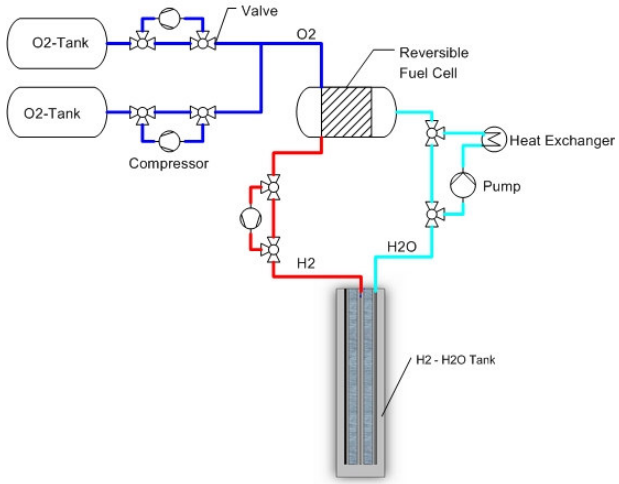

Figure 13. Schematic Process that combines both reversible oxygen and hydrogen storage and reversible fuel cell

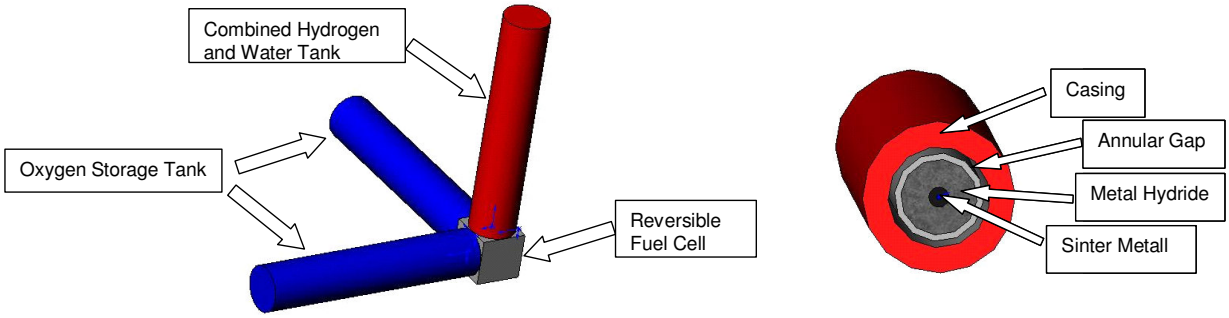

Figure 14. Multifunctional Structures for Hydrogen and Oxygen Storage

5. Reversible Hydrogen Storage

In order to arrive at an optimal tank structure, we decided to simulate the heat transfers and kinetic process during the hydrogen storage and release using ANSYS. The raw storage material is a mixture of NaH, and Al, subsequently referred to as "α Phase". Reaction with H creates the first loaded "β Phase", Na_3AlH_6. In the second reaction with H_2 the final "γ Phase" is created which is also the desired final state for optimal hydrogen loading.

$$I: NaH + \frac{1}{3}Al + \frac{1}{2}H_2 \leftrightarrow \frac{1}{3}Na_3AlH_6$$
$$II: \quad \frac{1}{3}Na_3AlH_6 + \frac{2}{3}Al + H_2 \leftrightarrow NaAlH_4 \qquad \text{(Eq. 1)}$$

The absorption and desorption of H_2 in metal hydride materials has been studied in detail by *Wang et al*[12]. Their model has been applied successfully in the simulation of the absorption processes in metal hydride materials[13] and has been fully applied in this simulation. Previous work[13] has focused mainly on tube-like geometries. These are advantageous in terms of construction effort, and variation of measures since pipes are readily available, and can basically be used as-is. In practice, however, more flexible geometries will be needed that can be fit to arbitrary available spaces on a spacecraft, or any other targeted vehicle.

This requirement is fulfilled by our innovative "cheese-hole" geometry as shown in **Figure 15**. It basically consists of an arbitrarily shaped closed parallel-plate vessel containing the Sodium Alanate.

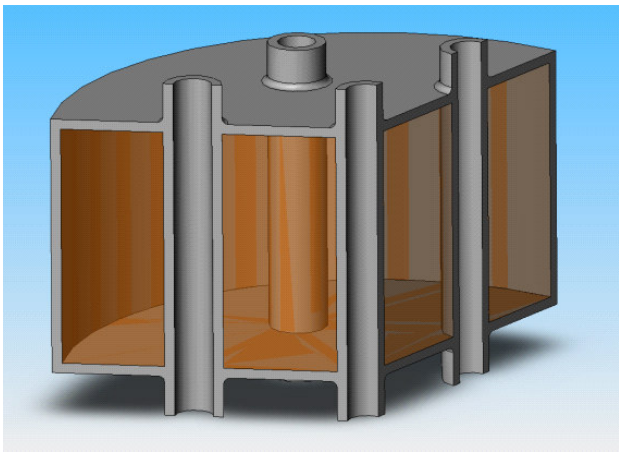

Figure 15. 120° pie slice of the adaptable geometry with interleaving cooling pipes. The Sodium Alanate core is shown in transparent orange.

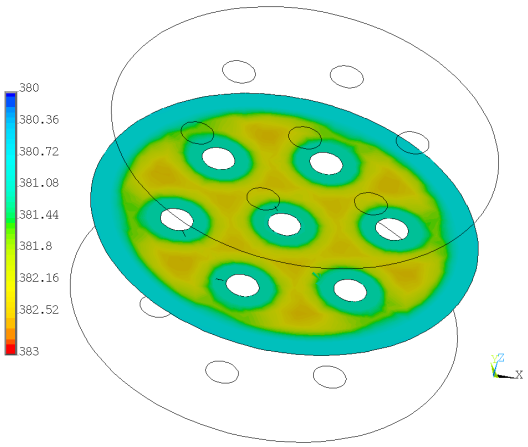

Figure 16. Temperature distribution in [K] on a XY-slice through the core. Time: 107.74 s

Cooling pipes are inserted in a regular pattern that does not principally depend upon the outer shape of the vessel. Hydrogen is exchanged through dedicated holes in the top and bottom plate (not shown). In this way the geometry can be adapted to a broad spectrum of applications, and is scalable in size. Our simulations show that the temperature distribution in that geometry is very homogenous with a temperature variation less than 2 K throughout the overall structure (see **Figure 16**). Therefore, we expect that such a tank system provides optimal hydrogen absorption and desorption as required for our reversible application. Future tests will target to verify and optimize our simulation results.

6. Reversible Oxygen Storage

Terrestrial fuel cells do not require oxygen tanks as they take out the oxygen from the ambient atmosphere. Space applications usually store oxygen as a compressed gas or generate oxygen with a solid gas generator. Both solutions are not easily reversible as the compressed tank requires a compressor that is large and heavy.

We are therefore developing a novel reversible oxygen solution based on a recently developed uncommon absorption/desorption behavior of a cation-stoichiometric cobalt oxide compound ($YBaCo_4O_7$) as recently reported by *Valldor* and *Andersson*[11].

In order to store oxygen in $YBaCo_4O_7$ several parameters must be maintained. The medium should be completely surrounded through an oxygen atmosphere and the temperature of oxygen as well as $YBaCo_4O_7$ must have a minimum temperature of 350°C in order for the absorption to take place. We intend to test the weight of our reactor prototype during the whole process and expect a rise of weight for the $YBaCo_4O_7$ compound of approximately 3 percent. This increase is equal to the absorbed mass of oxygen. Nitrogen will be used in order to rinse out the remaining oxygen in $YBaCo_4O_7$.

The test plant consists of two main parts: the heater part and the reactor part. The heater part is comprised of a tube and two covers. The tube performs two tasks: 1.) house the reactor with $YBaCo_4O_7$ and a spiral in which the gas is heated up and 2.) to receive a heating element on the outer surface which should guarantee a constant inside temperature of 350°C. The inside diameter of the tube is a few millimeters bigger than the diameter of the gas heating spiral. The length was chosen in order to give enough space for reactor and the gas-heating spiral. The tube can be closed on both sides with caps.

A schematic overview of the reactor test plant is illustrated in **Figure 17** and the reactor itself is detailed in **Figure 18**. With the first test results on the absorption/desorption capability, we will simulate and design the overall reversible oxygen tank system.

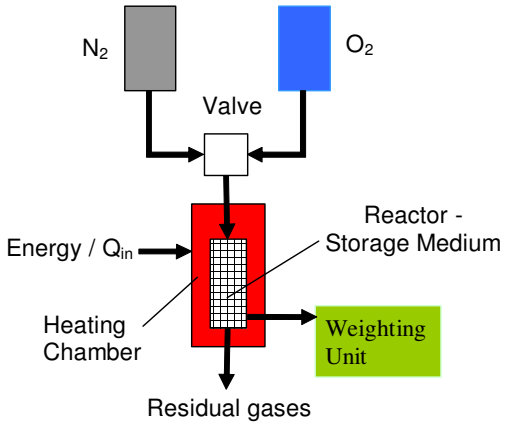

Figure 17. Flow process chart of Oxygen storage test plant

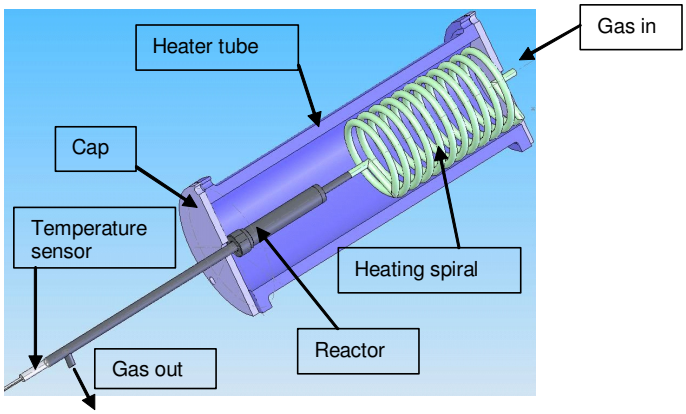

Figure 18. System arrangement

III. Micro Power Converters

The main expertise in our department is the development of micro propulsion systems for space applications including micro-rocket engine. Since the power density in such micro-combustion chambers is very large, it was natural to extend our efforts towards the development of micro power generators. Early in the project, a micro-turbine was developed to produce the power for the pumps of the rocket engine. This triggered our interest towards micro power converts in general since they may be used to increase the overall energy efficiency for a number of applications.

This chapter will give an overview of our micro-turbine development as well as their application towards a high-energy density cell in combination with our micro rocket engine.

C. Micro-Turbine Development

The exothermic reaction of the decomposition of hydrogen peroxide produces a relative hot mixture of water steam and oxygen. If this mixture is guided through a turbine, it can be used to generate electric power. From the various turbine types investigated in the past, the two with the most promising performance were manufactured. The first one has a rotor diameter of only 10 mm, whereas the second one has a diameter of 23 mm. The two types of rotors have different rotational speeds and thus different blade angles. The 10 mm diameter turbine has been manufactured using a LIGA process (Lithography, Electroplating, and Molding) in combination with an EDM process (Electrical discharge machining). It has been found that LIGA, when coupled with EDM, is the best technique to obtain rotors with small diameter and high aspect ratio blades[14]. The second prototype of the turbine rotor has been machined with a high –speed milling machine using aluminum as material for the turbine. The 23 mm diameter turbine has an optimum rotational speed of 80,000 instead of the 250,000 of the small turbine.

The achieved results after manufacturing and running test of the first turbine generation were not satisfactory. Collected experiences lead to an improved design for the second generation. Both designs are shown in **Figure 19** to express their differences in construction and solutions provided by improved design.

Several decisions were made in order to improve the power output of the turbine system. Analysis of the first generation turbine tests showed several possible reasons for the low power output including:

- Sealing problems
- Friction losses due to the bearing assembly
- Stray fields of the coupling magnets
- Inefficient magnetic coupling between turbine and generator

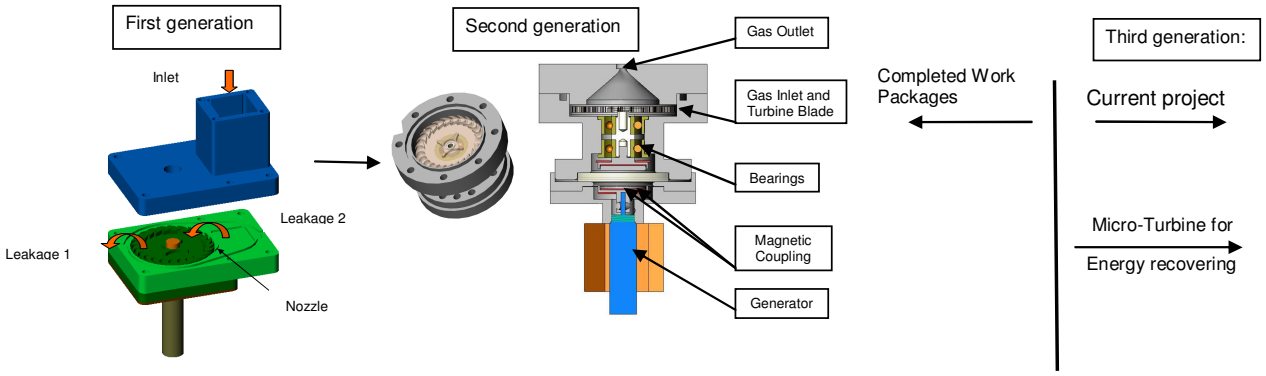

Figure 19. Micro Turbine progress

After precise consideration and analysis of turbine tests, several improvements on the third generation should be possible. The following list contains suggestions to improve the performance of the device:

- Use of stronger magnetic coupling or force a proprietary development
- New design of the turbine rotor blades
- An upgrade with stator blades
- Use of stronger generator
- Use of slide bearings

The use of oblong magnetic coupling gives higher torque transmission which should ensure that this coupling doesn't fail at high rotation speed. The last two generations weren't equipped with stator blades which are used to control the working fluid stream and guide it perfectly on the rotor blades.

The second generation turbine allowed higher power outputs at rotational speeds in the region of 10,000 to 15,000 rpm lower than in the previous design. A redesign of the gas inlet and outlet gave an improved performance for leakages. Application of two bearings instead one gives a better stabilization through the reduction of dynamics loads. Through an improved bearing assembly a smoother run of the turbine was observed. The third generation is currently under development. The first results are expected at the end of this year.

D. Micro Power Cells based on ARC Micro-Rocket Engine

Energy storage with high specific power densities is a key factor in present and future technology. ARC is developing miniaturized chemical propulsions systems using only green propellants (no toxicity, all propellants are available almost throughout the world) – see **Figure 20**.

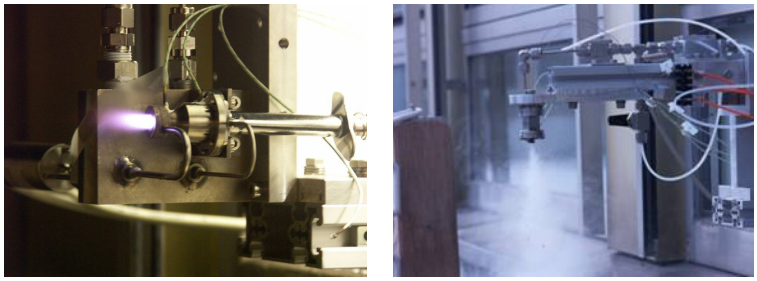

Figure 20. Mono- and Bi-Propellant µRocket Engine

In a monopropellant version, advanced catalyst beds are used with hydrogen peroxide (H_2O_2). In a bi-propellant version, Kerosene is used together with H_2O_2 in a micro combustor to achieve even higher power densities[15,16]. ARC is studying to transfer these micro-propulsion systems developed into high-power green energy cells with probably similar or even higher specific power and weight densities as Li-Ion batteries (see **Figure 22**) but with additional features such as:

- Immediate refill capability - fully recharge your energy cell within 10 seconds.
- Provide internal heat e.g. for high altitude UAVs.
- Provide clean water

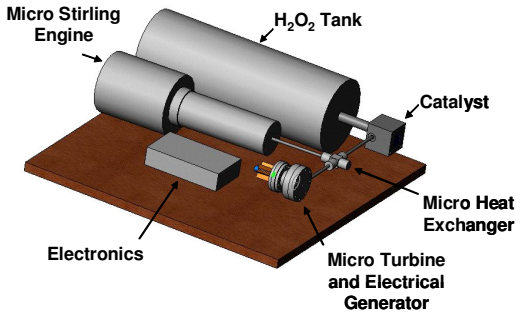

Figure 21. Micro Power Generator Prototype

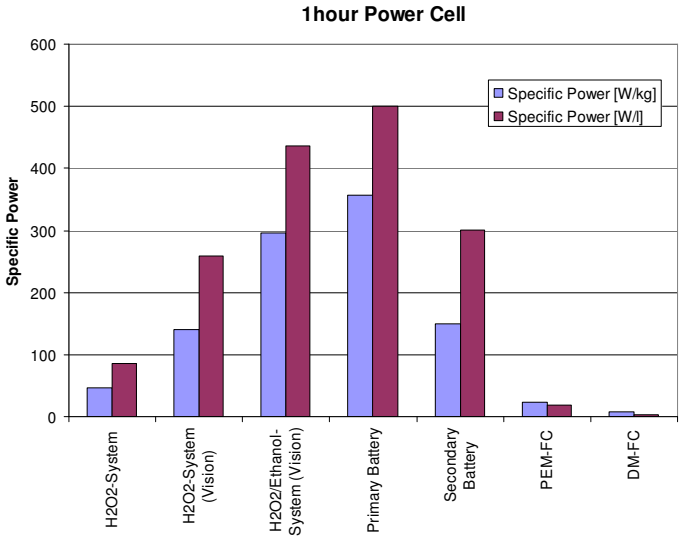

Figure 22. Micro-Power System Trade-Off

The basic components for such a generator are shown in **Figure 21**. A first generation (only H_2O_2 catalyst based) breadboard micro power generator with an optimized turbine and thermal design focusing on terrestrial and probably dual-use applications is currently under development. After system evaluation, also a second-generation combustion based system could be designed which promises even higher specific power densities.

IV. Conclusion

A wide variety of micro energy converters and hydrogen storage solutions is already under development at ARC that can be used for satellites – as well as an enabling technology for terrestrial applications.

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